DETERMINATIONS REGARDING THE ORGANOCHLORINE PESTICIDES CONTAMINATION IN MILK FROM BIHOR COUNTY

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ABSTRACT. The paper presents the results of the tests of the milk as raw material and consumption coming from some of the economic units in the Bihor county from the point of view of the contamination with organochlorine pesticides. Qualitative and quantitative determinations have been performed through the gas-cromatographic method using capillary columns and electrode with electrons capture. The values have been obtained through comparison with a complex standard diluted at 50 ppb. The tests submitted to verification consisted of lactic fat extracts purified through methods specific to determination in fields of ppm-ppb concentrations. The priority was the verification of the incidence of organochlorine compounds under sanitary – veterinary surveillance. After the determinations performed, we discovered, for most of the surveilled compounds, values under the approved limits, except for Endrine.

Keywords: organochlorine pesticides, consumption milk, cromatographic determinations

INTRODUCTION

The monitoring process of chemical contamination of food is an essential compound of food safety (Savu and Georgescu, 2004) and, according to Codex Alimentarius, supplies basic information about the identity of chemical contaminants for the selection of the priorities in what regards their follow-up and the limits admitted. Being a BASIC food, especially for children and older people (groups with major contamination risk), milk is a under sanitary-veterinary surveillance for the contaminant that can affect it, such as all kinds of pesticides. (23rd Order, 2007).If before the year 2000 in the Bihor county there was only a single trade company dealing with milk processing (that is "LUSO" Oradea – the Former Milk Processing Factory), nowadays their number is growing fast.

MATERIALS AND METHODS

MATERIAL

For the study that makes the object of the present paper, we have chosen some products obtained from shared stock trading companies that process cow milk as raw material gathered from the area and sells both consumption milk and various dairy products. They produce consumption milk with a fat content of 1,8% and 3.5%. We have chosen for the practical determination raw material milk and consumption milk, the 3.5% fat kind, because the contaminant being followed – organochlorine pesticides – are liposoluble.

Since this paper represents a research study and it is not the result of a control activity, we did not give the names of the companies whose products we investigated, but we coded them with letters RMA, RMB, RMC for raw material and CMA, CMB, CMC for consumption milk (table 5 and 6).

From geographical point of view, the tests have been taken from the middle area of the county (see Figure 1).

METHODS

The sampling of the products

The products have been taken directly from the unit and from the supermarkets. We have used as a guide the legislation specifications (SR EN ISO 707, 2002) for the sampling of the tests in what regards the recipients used, the minimum necessary quantities and transport conditions.

The recipients used were made of plastic (single use) and glass, with sealed tap. In order to remove all fat traces, the cleansing has been performed with hot water and detergents, the rinsing with hot water, washing with sulphochromic mixture, water rinsing and finally with distilled water. The recipients used for the organochlorine pesticides determinations have been rinsed with light petroleum as well. The recipients have been dried in the drying oven and kept sealed to prevent contamination. The tests have been transported in a freezing bag containing cooling bricks.

Chemical determinations connected to the composition, respectively fat content respected the due time-frame, that is 24 hours but the contaminants analysis have been performed both on fresh and frozen products since they took place at the Institute for Hygiene and Public Health from Cluj.

Tests preparation for the analysis

The preparation of tests for the analysis has been performed according to the legislation (STAS 6343, 1981). This aspect has as a purpose the homogenization of the sample and bringing it to the analysis temperature that is 20^0C.(Guş and Semeniuc, 2005).In

the case of frozen samples, they have been unfrozen at room temperature , after which we performed the same preparation operations as in the case of fresh products (only for fat separation for the determination of pesticides).

Test preparation for cromatography

Since we analise the lyposolubile contaminants, the phases to prepare the samples for qualitative and quantitative determinations are:

Separation the the fat that contains the toxic product: the method we used was partititon n-hexan/acetone, drying on anhidre sodium sulphate and evaporation of the solvent at low pressure (SR EN 1528/1and /2, 2004), according to the scheme shown in table 1.

Fig. 1 Geographical area of sampling

Table 1

EXTRACTION OF FAT WITH TOXIC PRODUCT

Nr.	Operation performed	Devices / Glassware
1	• 100 ml milk • 500 ml n-hexan-acetone = 2:1 • Homogenization 4 minutes • Separation phases	Glass 1000 ml
2	• Elutriation organic layer over 500 ml solution of 2% Na_2SO_4	Separation funnel
3	• 50 ml an-hexan/acetone = 2:1	Glass 1000 ml from (1)
4	• Elutriation organic layer	Separation funnel (2)
5	• Stirring the funnel for 30 sec • Removal of the water part	Separation funnel
6	• 500 ml solution 2% Na_2SO_4 • Removal of the water part	Separation funnel
7	• Filtering of the organic layer over 20 g Na_2SO_4 in balloon	Filtering funnel with glass frit Evaporator balloon
8	• Evaporation at 50 ^{0}C, low pressure • Final evaporation, nitrogen draft	Spinning evaporator

Table 2

PURIFICATION OF FAT WITH TOXIC PRODUCT

Nr.	Executed operation	Devices / Glassware
	Faza de partiţie	
1	• Max 3 g molten fat + petroleum ether (total max volume 15ml) • 30l saturated acetonitril with petroleum ether, • Strong stirring 1 min	Separation funnel 125 ml (1)
2	• The acetonitryl layes is dripped over: • 650 ml water + 40 ml nacl saturated solution + 100 ml petroleum ether	Separation funnel 1000 ml (1)
3	• The ether layer from the 125 ml funnel is extracted 3 times x 30 ml saturated acetonitril with petroleum ether in: • Strong stirring, separation 1 ml	Separation funnel 125 ml (1)
4	• All the extracts at phase 3 are combined in: • Shaking in horizontal position for 30-45 sec	Separation funnel 1000 ml (1)
5	• The water phase passes in:	Separation funnel 1000 ml (2)

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	• +100 ml petroleum ether, strong shaking 15 sec • - removal of the water	
6	• The ether layer from the 1000 ml (2) funnel combines with the one in the 1000 ml funnel (1) • Washing with 2 x 100 ml water, removal of the water layer	Separation funnel 1000 ml (1)
7	• The etherit layer is passed through Na_2SO_4 column, anhidre : • Rinsing funnel and column with 3 x 10 petroleum ether	Cromatographic column d_e x L = 25x50mm PTFE tap otton glass Seal
8	• Collection etheric extract combined in the evaporator : • Evaporation up to 10 ml	Kuderna Danish, 500 ml, lined tube
	Cromatograph phase	
1	• Column preparation : • 10 cm activated Florisil (after ramming) • 1 cm Na_2SO_4 anh	Cromatographic glass column: - di x L = 22 x 300 mm - PTFE Tap - Frit glass or glass wool tap
2	• Humidification column with 40÷50 ml petroleum ether • The solution is passed through column at point 8, Q_{max} 5 ml/min) • Recipient wash with 2 x 5 ml petroleum ether → column • Tube's walls washing with a little petroleum ether → column	Florisil column 500 ml K-D balloon with liner tube, under column (1)
3	• Eluation with: • 200ml mixture A petroleum ether / ethylic ether = 94/6 (V /V) • Speed: 5 ml/min (all POC without dieldrine and endrine	Florisil column Collecting balloon (1)
4	• Eluation with : • 200ml mixture B petroleum ether / ethylic ether = 85/15 (V /V) • Speed 5 ml/min (dieldrine and endrine)	Florisil column Collecting balloon (2)
5	• Separate evaporation of the eluates from 3 and 4 un to approx. 5 ml	Spinning evaporator

Purification of the extract was performed through partitioning with acetonitril/petroleum ether and passing through the column with activated Florisil (SR EN 1528-3, 2004), according to the scheme shown in the table 2.

Density determination

The measurement of the density of milk was necessary for the determinations in which we worked with test measured by volum but where, in the calculus, we used quantities. We applied the aerometric method (STAS 6347, 1973) using a thermolactodensimeter, that allowed us to correct the read values according to the temperature of the sample for values at 20^0C.

Milk tests have been measures as volume (100 ml) and we determined the density (corrected to the measured temperature of the test) both for calculation necessity and for stressing the fact that we have worked with a quantity of approximately 100g product, as required by the used method . The concrete values can be found on the calculation sheet of each sample. There is an example is shown in Annex 1 of the paper.

The determinations of the residues of organochlorine pesticides in the studies products

We have used the method most recommended by specialised literature (Bara et al, 1998; Hura 2006).

Guidelines on Good Laboratory Practice in Pesticide residue Analysis, 1993), as set forth by current legislation for the calculation of pesticide residues in dairy products. It is the gas chromatography (SR EN 1528-4, 2004).

Used Devices

We have used a GC 2010 Shimadzu gas chromatograph, with the following characteristics :

- Capilar column type RTX -CL- pesticides 30 m length an 0,25 mm diameter. The column works at a temperature between 150 ÷ 320^0C with a gradient ofe 3^0C/2 minutes
- Electrode with electrons capture (ECD), nuclid ^{63}Ni – 370 MBq (10mCu)
- Autosampler injection system with 6+2 spaces for vials, type AOC-210

Cromatography conditions :

- Injection temperature (splitting) = 250^0C
- Splitting temperature = 163,5 Kpa
- Splitting gas : He with a 124 ml/min flow at scavenging 30 ml/min
- Carrying gas : N_2 ultrapure 99,99%
- Detector : = 320^0C, detector current of 2 nA, make-up flow = 30 ml/min

The device is connected to a computer and used a specific program for the interpretation of the results. Cromatograms are displayed on a singular monitor for the tested sample or together with the chromatogram of the used sample. The program supplies the retention times, the height of the picks and their surface, through automatic integration.

Qualitative and quantitiative calculations

For the qualitative and quantitative calculation of the contaminants possibly present in the tested products, we used a standard produces by the RESTEK company No 32292, Lot nr A021837, type "Organochlorine pesticide Mix AB ≠2" having a concentration of 200 ppb.

Table 3 shows the elution order of the components of the standard mixture used. Notice the fact that the standard contains, with one exception (hexachlorobenzen) all the organochlorine compounds under sanitary-veterinary surveillance in vegetal and animal foods, therefore in milk and dairy products.

Milk tests submitted to the verification from the point of view of the contamination with organichlorine pesticides have undergone the procedures explained in "Test preparation for chromatography" part of the paper. The fat extract purified, retaken in petroleum ether was submitted to the chromatography under the same conditions as the standard test, as well as the blanks-test of the used reagents, according to the separation / purification method.

Table 3

COMPOSITION USED STANDARD

A	B	C	D	E
1	Alpha HCH	Alpha HCH		6.811
2	Gamma HCH	Gamma HCH		7.978
3	Beta HCH	Beta HCH		8.403
4	Delta HCH			9.074
5	Heptachlor	Heptachlor	The sum of heptaclor and heptaclor-epoxid expressed in heptachlor	9.898
6	Aldrine	Aldrine	Aldrin alone or combined, expressed in dieldrine	11.156
7	Heptaclhor epoxide			14.036
8	Gamma Chlordan	Chlordan	Chlordan (the sum in isomers cis and trans and oxichlordan	14.657
9	Alpha Chlordan		expressed in chlordan	15.322
10	4,4' DDE			15.849
11	Alpha endosulphan	Endosulphan	Sum of α and β and sulphate endosulphane expressed in endosulphane	16.088
12	Dieldrine			17.103
13	Endrine	Endrine		18.18
14	4,4' DDD			19.105
15	Beta Endosulphan			19.361
16	4,4' DDT	DDT	Sum of DDT, DDE and DDD isomers expressed DDT	20.564
17	Aldehide Endrine			21.585
18	Metoxichlor			23.648
19	Endosulphan sulphate			23.845
20	Endrine cetone			25.205

***A.** Elution order; **B.** Type of organochlorine compound in the standard; **C**. Type of organochlorine compound surveilled from a Sanitary – Veterinary perspectiv; **D.** Observations from the Sanitary-Veterinary surveillance program; **E**. Retention time for the standard's compounds*

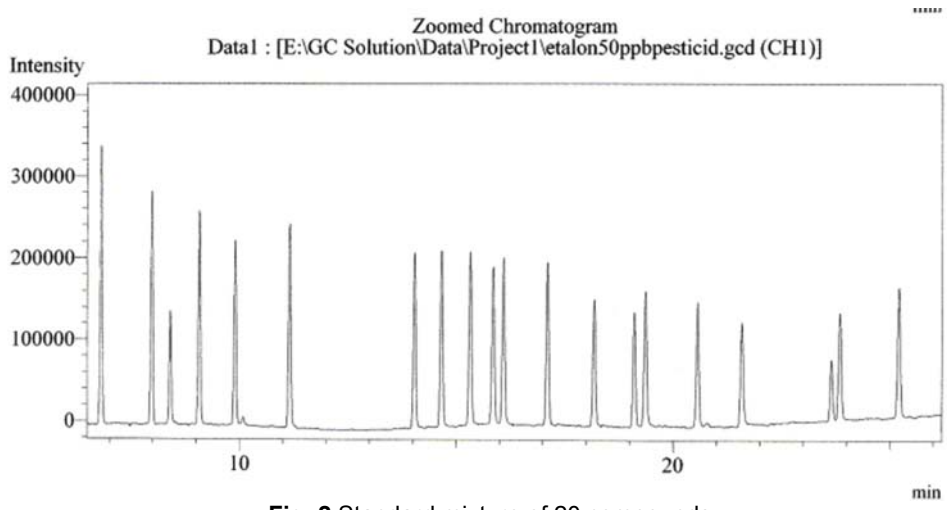

Fig. 2 Standard mixture of 20 compounds

The figure 2 shows the chromatogram of the standard mixture at 50 ppb concentration

For the quantitative determinations, we have used the value of the surface of the picks (compounds that have been previously identified as being present in the tested sample). In order to prevent possible calculation errors, we introduced the data we obtained through the automatic integration of the unknown tests and the used standard in EXCELL calculation sheets, selecting only the surface of the picks we need, that is the compounds that were found from the qualitative point of view.(Annex 1 of the paper)

In the quantitative calculation, we took into consideration the percent of recovery of the residues that we calculated working in the same conditions as for the chromatography of the tested samples, but we worked on some separate fat samples to which we added a quantity determined by the sample. The quantitative calculations are possible only if the recovery level for each tested compound is between 70 ÷110%(SR EN 1528/4, 2004) This is what we have accomplished during the tests, which can also be seen in the results section. In table 4 we have mentioned the values of the recovery degree for the compounds that could be effectively found in the tests.

Table 4

RECOVERY DEGREE, R – STANDARD 50 PPM

Organochlorine compound, found in the test	Alpha HCH	Gamma HCH	Beta HCH	Alpha endosulphan	Endrine	4,4' DDT
R, standard 50 ppm	87,2	87,3	91,2	94,0	104,7	92,4

RESULTS

For the qualitative determination, we compared the retention times for the significant picks that appear on the chromatogram of the unknown tests with the ones of the compounds from the standard sample. This way, we can determine which of the compounds that are present in the standard can also be found in the tested sample (Gocan, 1998). The results are written in table 5.

The result of the quantitative calculation of organochlorine pesticides residues is shown in table 6.

The figure 3 shows as an example the chromatogram of one of the studied sample (RMC).

Table 5

DRINKING MILK 3,5%, QUALITATIVE DETERMINATION OF THE ORGANOCHLORINE PESTICIDE RESIDUES

A	B	C	D					
			RMA	RMB	RMC	CMA	CMB	CMC
1	Alpha HCH	6.811	6.951	6.798	6.799	6.799	6.798	6.799
2	Gamma HCH	7.978	7.956	7.963	7.963	7.969	7.963	7.973
3	Beta HCH	8.403	8.380	8.387	8.380	8.380	8.385	8.380
4	Delta HCH	9.074						
5	Heptachlor	9.898						
6	Aldrine	11.156	11.163					
7	Heptaclhor epoxide	14.036						
8	Gamma Chlordan	14.657						
9	Alpha Chlordan	15.322						
10	4,4' DDE	15.849						
11	Alpha endosulphan	16.088	16.050	16.060	16.060	16.072	16.062	16.061
12	Dieldrine	17.103	17.057					
13	Endrine	18.18	18.159	18.168	18.169	18.168	18.168	18.167
14	4,4' DDD	19.105	19.064					
15	Beta Endosulphan	19.361						
16	4,4' DDT	20.564	20.520	20.531	20.533	20.550	20.540	20.525
17	Aldehide Endrine	21.585						
18	Metoxichlor	23.648						
19	Endosulphan sulphate	23.845						
20	Endrine cetone	25.205						

***A**. Eluation order; **B.** Type of organochlorine compound in the standard; **C.** Retention time in the standard; **D.** Retention time in the test*

Table 6

THE CALCULATION OF THE ORGANOCHLORINE PESTICIDE RESIDUES IN DRINKING MILK SAMPLES (06.08.2007)

Nr crt (*)	Organochlorine compound found in the sample	Max. allowed concentration Ppm	Concentration found in the test ppm					
			RMA	RMB	RMC	CMA	CMB	CMC
1	Alpha HCH	0,004	0,0002	0,0002	0,0006	0,0002	0,0002	0,0005
2	Gamma HCH	0,003	0,0007	0,0007	0,0005	0,0008	0,0008	0,0004
3	Beta HCH	0,008	0,0004	0,0009	0,0002	0,0011	0,0008	0,0002
6	Aldrin (HEOD)[1]	0,006	0,0001					
12	Dieldrine	0,006	Sld					
11	Endosulphan [2]	0,004	0,0015	0,0016	0,0013	0,0023	0,0015	0,0012
15								
19								
13	Endrine	0,0008	0,0006	0,0008	0,0006	0,0007	0,0005	0,0005
10, 14, 16	DDT [3]	0,04	0,0008+ 0,0001= 0,0009	0,0011	0,0008	0,0011	0,0013	0,0011

** - refers to the eluation order of the compounds in the samples and the standards, respectively; 1- alone or combined, expressed in dieldrine; 2 - sum of α and β and endosulphan sulfat expressed in endosulphan); 3 - sum of DDT, DDE and DDD isomers expressed in DDT*

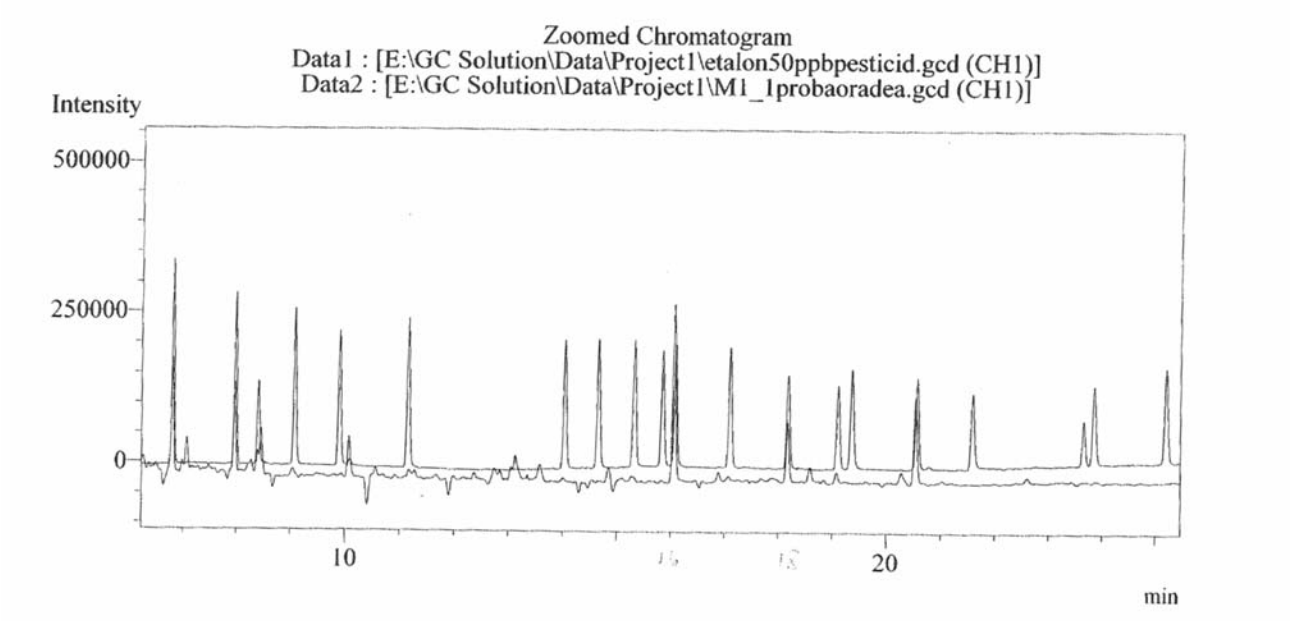

Fig. 3 Chromatogram of one of the studied sample (RMC)

DISCUSSIONS

The interpretation of the statistic data that we analysed before the drawing up of the present report (Chiş Adriana, 2007), shows, at first sight, a reduced incidence of food contamination with organochlorine pesticides starting with the year 2000. But if we take into consideration the extremely reduced number of tests in the monitoring programs, linked with the increasing number of the food processing units appeared in the last three years, we considered that a research focused on a single type on a precise geographic area, can offer a deeper result.

A. Regarding the milk tests verified in the programs of sanitary-veterinary surveillance The number of samples taken each year and submitted to verification for detecting pesticide residues in food and the one referring to milk and dairy products is far from covering the range of dairy products produced and sold in our county, as can be seen in table 7.

Table 7

MILK TESTS / TOTAL SAMPLES

Food type	Year													
	2000		2001		2002		2003		2004		2005		2006	
	*	**	*	**	*	**	*	**	*	**	*	**	*	**
Milk and dairy	12	-	5	-	30	1	5	-	8	-	23	-	12	-
TOTAL food products	57	-	20	-	93	2	48	1	49	-	65	1	47	1
% Dairy from total samples	21	-	25	-	33	50	10	-	16	-	35	-	25	-

** - total number of taken samples; ** - of which positive*

The gathering and verification of milk samples, oncentrated in time and space, allows a deeper evaluation of the contamination potential with residues of organichlorine pesticides, as seen from the data taken in table 5 and 6.

B. Regarding the types of organochlorine pesticides followed up from a sanitary-veterinary point of view and whose residues appear in the tested samples of milk (Table 5**).**

The pesticides from group HCH (isomers α, β, γ) appear in all the tested samples but they are way under the maximum admitted values;

Aldrin, Dieldrin and 4,4' DDD pesticide types appear randomly and with values under the maximum admitted values, even under the detection limit (Dieldrine)

The pesticides that are found in all tested samples, except from the HCH isomers group are:

- Endosulphan
- Endrine
- DDT (alone as isomer 4,4' or together with 4,4'DDD)

The motivation of this situation can be based upon the following considerations:

- The endosulphan is the only organochlorine pesticide still in use în the European Community countries (EFSA 2006)

The endrine, stereoisomer of Aldrine and eldrine has a great capacity of persistence and accumulation in the soil
(Kegley et al., 2007)

- DDT and the other substances from the same class have been used at large scale and, despite the fact they have not been used in a very long time, they can still be found in all environment factors therefore, in food as well due to its high-bioaccumulation capacity and the halving time.

So, it is obvious the necessity of the verification of the residues from pesticides in the animals' food and in the water from the grassing areas because the food chain is the most responsible for the food pollution with pesticides.

C. Regarding the concentration of organochlorine pesticides found in the studies samples (table 6)

We found values equal with MRL (maximum residues level) for :

- Endrine: 1 test of drinking milk (0,008 ppm);

Values close to MRL

- Endrine: all the other tests show values close to MRL (maximum residues level), that is between 0,005÷0,007 ppm

Values of the same size order as MRL but lower

- Endosulphane : values between 30 % ÷ 57,5% of MRL

Significantly different values (size order) towards MRL

- HCH, Aldrin, Dieldrin, DDT isomers

CONCLUSIONS

As a final conclusion, we consider that it is necessary to continue the studies in different periods of times and using dairy products with a high level of fat (cream, cheese) in order to realise statistic studies relevant in the field of contamination of dairy products with organochlorine pesticides.

We also consider that it is important to continue the monitoring program with a significant increase of the number of samples to be studies. This aspect is also tied to the funds, since we all know that this kind of tests are expensive and complicated.

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ANNEX - CALCULATION SHEET FOR THE OCP CONCENTRATION AT SAMPLE RMC

50 ppb standard

Nr. crt.	Organo-chlorine compound From standard LC	Retention time Standard	Retention time sample	Area of the interest pick	Corresponding surface From the blank test	Corrected area Ac	Corresponding Area from the standard	Sample mass g	Sample conc ppb	vol ml	Ca x C / Standard area	Pesticide Conc. ppb	Pesticide Conc. ppm	R %	PPM corrected
1	***alfa HCH***	6.811	6.799	481615.3	27996.5	453618.8	1087544	102.9	50	2	20.8552	0.40527	0.0004	87.2	**0.0005**
2	***gama HCH***	7.978	7.973	325780.9	19249.2	306531.7	929044.8	102.9	50	2	16.4971	0.32058	0.0003	87.3	**0.0004**
3	***beta HCH***	8.403	8.38	73655.6		73655.6	454994.8	102.9	50	2	8.09411	0.15729	0.0002	91.2	**0.0002**
4	delta HCH	9.074					867166.7								
5	Heptaclor	9.898					791830.3								
6	Aldrin	11.156					931809								
7	Heptaclor epoxid	14.036					927627.4								
8	gama Chlordan	14.657					842111.9								
9	alfa Chlordan	15.322					851479.3								
10	4,4' DDE	15.849					780367.4								
11	***alfa endosulphan***	16.088	16.062	1044232	129688.9	914542.6	808477.9	102.9	50	2	56.5595	1.0991	0.0011	94.0	**0.0012**
12	Dieldrin	17.103					804192.7								
13	***Endrine***	18.18	18.167	335884.9	10398.1	325486.8	656978.6	102.9	50	2	24.7715	0.48137	0.0005	104.7	**0.0005**
14	4,4' DDD	19.105					578571.8								
15	beta Endosulphan	19.361					709807.6								
16	***4,4' DDT***	20.564	20.525	671098.2	18339.6	652758.6	628075.8	102.9	50	2	51.965	1.00981	0.0010	92.4	**0.0011**
17	Endrine aldehide	21.585					561882.2								
18	Metoxichlor	23.648					295236.6								
19	Endosulphan sulphate	23.845					575368.4								
20	Endrin cetone	25.205					*696551.6*								

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